



Attorney Docket No.: 0492611-0543/MIT-9277CON 1

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

Applicant: Seleznev, *et al.* Examiner: Cooke
Serial No.: 10/799,436 Art Unit: 1751
Filing Date: March 12, 2004

Title: VACUUM PROCESSING FOR FABRICATION OF
SUPERCONDUCTING THIN FILMS FABRICATED BY METAL-
ORGANIC PROCESSING

Commissioner for Patents
P. O. Box 1450
Alexandria, VA 22313-1450

Declaration of Michael J. Cima under 37 C.F.R. 1.132

I, Michael J. Cima, declare as follows:

1. I am the Sumitomo Electric Industries Professor of Engineering at the Massachusetts Institute of Technology (MIT). My CV is attached as Exhibit 1.
2. I am an inventor of U.S. Patent Application No. 10/799,436, entitled "Vacuum Processing For Fabrication of Superconducting Thin Films Fabricated by Metal-Organic Processing," filed March 12, 2004.
3. I have reviewed the specification and claims of the above-referenced patent application as well as the Office Action mailed March 29, 2005, and understand that the Examiner requires further information in support of the claims directed to methods of forming YBCO films having resistivities between 100 and 600 $\mu\text{Ohm-cm}$ and/or critical current densities of 0.1 MA/cm² at zero field.
4. On or about April, 2002, American Superconductor Corporation, the licensee of this application, supplied us with green yttrium-barium-copper oxyfluoride films on nickel tape. I am informed that those samples were 0.8 μm thick, 10 mm wide, and 20 mm long. They asked us to process these samples under the conditions described in Exhibit 2. Samples were placed in a furnace and the temperature ramped up to 785°C over 2 minutes (see column labeled "Heating"). Samples were annealed at 785°C in a mixture of nitrogen, oxygen and water vapor, with the partial pressure of water vapor held at the pressure in the "Low" column and then the

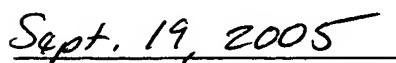
pressure in the "High" column for the times indicated in the column labeled "Time." The total pressure in torr is given in the column labeled P. The atmosphere was 10% oxygen except for the last sample, D15R48, for which the atmosphere was 1 torr oxygen, with no nitrogen added. The samples were annealed under oxygen at 475°C. The measured resistance and critical current density (self field) and calculated resistivity are as shown.

5. In conclusion, heat treating samples using the techniques described above results in YBCO films having resistivities between about 300 $\mu\text{Ohms-cm}$ and about 800 $\mu\text{Ohms-cm}$, for example, between about 300 $\mu\text{Ohms-cm}$ and about 600 $\mu\text{Ohms-cm}$, and critical current densities of about 0.1 MA/cm² or greater measured at zero field.

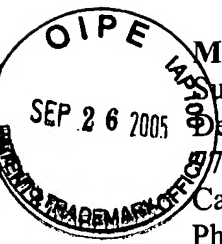
6. I, Michael J. Cima, declare that all statements made herein of my own knowledge are true and that these statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful, false statements and the like are made punishable by fine or imprisonment, or both, under §1001 of Title 18 of the United States Code and that such willful, false statements may jeopardize the validity of the application or any patents that may issue thereon.



Michael J. Cima



Date



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Education:

B.S. Chemistry (1982) / Ph.D. Chemical Engineering (1986), University of California at Berkeley

MIT appointments:

Professor with tenure Department of Materials Science and Engineering
Director, Ceramics Processing Research Laboratory
Materials Processing Center Investigator
Center for Biomedical Engineering Investigator

External appointments

Co-Founder and Director of MicroChips Inc., a developer of microelectronic based drug delivery systems.

Director of Ferrite Components Inc., a manufacturer of high power RF components and systems

Senior consultant to Transform Pharmaceuticals Inc. and is a member of its management team. TPI is a developer of high throughput screening technologies for pharmaceutical development

Senior consultant to ETEX Corp. a manufacturer of calcium phosphate based cements for orthopedic applications and drug delivery

Other significant consulting relationships include expert reviews concerning pharmaceutical formulation and processing.

Biosketch / Research Overview:

Dr. Michael J. Cima is a Professor of Materials Science and Engineering at MIT. He earned a B.S. in chemistry in 1982 (phi beta kappa) and a Ph.D. in chemical engineering in 1986, both from the University of California at Berkeley. He received the Norton chair at MIT in 1988 in the Materials Science and Engineering Department as an Assistant Professor. Prof. Cima became Director of the Ceramics Processing Research Laboratory in 1989 and received a tenured appointment from MIT in 1992. He was promoted to full Professor in 1995. He was elected a Fellow of the American Ceramics Society in 1997 and has recently been awarded the Sumitomo Electric Industries Chair at MIT. Prof. Cima is author or co-author of over one hundred and eighty scientific publications, twenty-seven patents, and is a recognized expert in the field of materials processing. He has been the principle investigator on several multimillion-dollar Defense research projects and many other sponsored research programs at MIT. Prof. Cima's recent research is primarily in four areas: advanced forming technology, ceramic thin film processing, MEMS devices for medical electronics and drug delivery, high-throughput

development methods for formulations of materials. Forming methods for complex macro and micro devices are of particular interest including MIT's three-dimensional printing process. His research group is one of the leaders in the development of chemically derived epitaxial oxide films for HTSC coated conductors. They are also leaders in the field of particle-particle and particle-surface interactions. He and collaborators are developing implantable MEMS devices for unprecedented control in the delivery of pharmaceuticals. Most recently, his group has been working on methods to speed the development of materials systems that are based on complex formulations. Finally, through his consulting work he has been a major contributor to the development of a high throughput system for discovery of novel crystal forms of pharmaceuticals.

Professional Affiliations

American Chemical Society, Materials Research Society, American Ceramic Society, Ceramic Society of Japan

Honors

Fulrath Award 2003

Winner of the International Award of Materials Engineering for Resources, 1998

Sumitomo Electric Industries Chair, 1997

Fellow of the American Ceramics Society, 1997

R&D 100 Award for 3DP Applied to Metal Casting, 1994

ALCOA Foundation Science Award, 1991

Extractive Metallurgy Award, TMS, best paper, 1990

Norton Chair, 1989

Phi Beta Kappa, UC Berkeley, 1982

Interests:

Processing Science and Engineering

Pharmaceutical processing

Drug delivery technologies

MEMS

Rapid Prototyping Technology

3D Printing

Chemical and Physical Phenomena in Powder Processing

1. Cima, M. J. and Rhine, W. E., "Powder Processing for Microstructural Control in Ceramic Superconductors," *Advanced Ceramic Materials*, 2, 329-36, (1987).
2. Cima, M. J. and Lewis, J. A., "Firing-Atmosphere Effects on Char Content from Alumina-Poly(Vinyl Butyral) Films," *Ceramic Transactions*, 1, 567-74, (1988).
3. Cima, M. J., Rhine, W. E., and Bowen, H. K., "Synthesis and Processing of Ceramic Superconductors," (1988).
4. Cima, M. and Brewer, L., "The Generalized Lewis Acid-Base Titration of Palladium and Niobium," *Metallurgical Transactions B: Process Metallurgy*, 19B, 893-917, (1988).
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6. Cima, M. J., Chiu, R., and Rhine, W. E., "Barium Yttrium Copper Oxide ($\text{Ba}_2\text{YCu}_3\text{O}_{6.9}$) Powder Preparation by Sol-Gel Emulsion Techniques," *Materials Research Society Symposium Proceedings*, 99, 241-4, (1988).
7. Peterson, S. C. and Cima, M. J., "Magnetic Inducement of Texture in Barium Yttrium Copper Oxide ($\text{Ba}_2\text{YCu}_3\text{O}_{6.9}$) Particle Assemblies under Cryogenic Conditions," *Journal of the American Ceramic Society*, 71, C458-C459, (1988).
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54. Kagan, C. R. and Cima, M. J., "Solution Precipitation of Cadmium Selenide Quantum Dots," *Materials Research Society Symposium Proceedings*, 283, 841-6, (1993).
55. McIntyre, P. C. and Cima, M. J., "Microstructural Evolution During Epitaxial Growth of Chemically Derived Barium Yttrium Copper Oxide ($\text{Ba}_{2-y}\text{Cu}_{3+7-x}$) Thin Films," *Materials Research Society Symposium Proceedings*, 280, 371-4, (1993).
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Invited Lectures

- Sept. 1989 "Microstructural Development in Chemically and Particulate $\text{Ba}_2\text{YCu}_3\text{O}_y$ Films," DARPA contractor meeting Santa Barbara CA.
- Nov. 1989 "Directional Solidification of High-Temperature Superconductors," AICHE Topical Conference on Emerging Technologies in Materials San Francisco CA.
- Dec. 1989 "Liquids and Processing of High Temperature Superconductors," National Chemical Laboratory for Industry Tsukuba Japan.
- Dec. 1989 "Liquids and Processing of High Temperature Superconductors," University of Tokyo Tokyo Japan.
- Feb. 1990 "Centrifugal Casting of Ceramic Components," Third International Conference on Powder Processing Science San Diego CA.
- March 1990 "Percolation Phenomena in Ceramic Processing," Sixth Annual Symposium on Advanced Ceramics American Ceramic Society, Hudson-Mohawk Section Troy N.Y.
- March 1990 "Chemical Issues in Contemporary Ceramics Processing," SUNY Binghamton NY.
- April 1990 "Whisker Percolation in Ceramic Matrix Composites," Annual Meeting of the American Ceramics Society Dallas TX.
- April 1990 "Three Dimensional Printing: Forming of Ceramic Structures for Casting," Annual Meeting of the American Ceramics Society Dallas TX.
- April 1990 "The Removal of Processing Aids," Annual Meeting of the American Ceramics Society Dallas TX.
- July 1990 "Particulate Ceramic Processing," Gordon Conference on High Temperature Chemistry Kimball Union Academy Meriden NH.
- Sept. 1990 "Processing of Particulate Ceramics," Barnett F. Dodge Lecturer Department of Chemical Engineering Yale University New Haven CT.
- Nov. 1990 "The Removal of Processing Aids from Ceramic Greenware," Annual Meeting of the American Institute of Chemical Engineers Chicago IL.
- Nov. 1990 "Centrifugal Casting of Ceramic Components," Annual Meeting of the American Institute of Chemical Engineers Chicago IL.
- Jan. 1991 "Metalorganic Deposition of High- J_c $\text{Ba}_2\text{YCu}_3\text{O}_{7-x}$ Thin Films on Single Crystal Substrates"; XII Winter Meeting on Low Temperature Physics Valle de Cuernavaca Morelos Mexico.
- March 1991 "Drying Behavior of Ceramic Granular Films," 4th International Conference on Ceramic Powder Processing Science Nagoya Japan.

- May 1991 "Directional Solidification of HTSC Oxides" The Third ISTEC Workshop on Superconductivity "New Superconductors and Syntheses," Kumamoto Japan.
- Oct. 1991 "Semisolid Solidification of $\text{Ba}_2\text{YCu}_3\text{O}_{6+y}$ Fibers," 4th International Symposium on Superconductivity Tokyo Japan.
- Nov. 1991 "Enabling Process Technologies for Ceramics," International Journal of the Society of Materials Engineering for Resources Vol. 1 No. 1 pp. 3-9 (1993) Akita City Japan.
- Dec. 1991 "Drying of Granular Ceramic Films," Materials Research Society Boston MA.
- Feb. 1992 "Ceramics Processing: Structure Form and Performance," Florida Advanced Materials Chemistry Conference Palm Coast FL.
- April 1992 " $\text{Ba}_2\text{YCu}_3\text{O}_{6.5}$ Solidification Kinetics from Semisolid Melts," Workshop on "Bulk Processing and Critical Current Density of High- T_c Superconductors," Argonne National Laboratory Argonne IL.
- June 1992 "Ceramics Processing in the 1990's" A.R. Cooper Distinguished Speaker Award at the 19th Annual Symposium of the Northern Ohio Section of the American Ceramic Society Cleveland OH.
- July 1992 "Construction of Computer-Derived Microstructures from 3D Printing," Solid Freeform Fabrication Symposium Austin TX.
- Nov. 1992 "Enabling Technologies in Ceramics Processing," Departmental Colloquium Speaker Department of Materials Science and Engineering University of Illinois at Urbana-Champaign Urbana IL.
- Nov. 1992 "Morphology of BaY_2CuO_5 and Semisolid Solidification of $\text{Ba}_2\text{YCu}_3\text{O}_{6.5}$," Fall Meeting of the TMS Society Chicago IL
- Jan. 1993 "Challenges in Solid Free Form Fabrication," ONR Manufacturing Science Workshop Chantilly VA.
- Feb. 1993 "Ceramics Process Technologies for Micro and Macro Control," Departmental seminar Department of Materials Science and Engineering University of Connecticut.
- Sep. 1993 "3D Printing: Computer-Derived Shapes and Structures," Raytheon Lexington MA
- July 1993 "Inhomogeneity and Anisotropy of Tape Cast Ceramic Films used for Multilayer Ceramic Structures," Workshop on Ceramics Science and Technology Ankara Turkey
- Oct. 1993 "Critical Current Enhancement of Directionally Solidified HTSC Materials: Microstructural Control," Engineering Research Symposium Tokyo Electric Power Corp. Tokyo Japan.
- Nov. 1993 "Drying and Binder Removal from Ceramic Components," AIChE Annual Meeting St. Louis MI.

- Jan. 1994 "Low Cost High Performance Tooling by 3D Printing Ceramic Injection Molds from Computer Files," 18th Annual Conference and Exposition On Composites Materials and Structures Cocoa Beach FL.
- Jan. 1994 "Enabling Technologies and Science for Powder Processing: Structure Form and Performance" Departmental seminar Department of Ceramic Engineering Rutgers University Piscataway NJ
- Apr. 1994 "Creation of order in Nanocrystalline Solids by Ion beam assisted deposition" Workshop on Nanoparticle Processing University of Illinois at Urbana-Champaign Urbana IL
- Apr. 1994 "Density and Anisotropy of Tape-Cast Ceramic Green Sheets," Synthesis Processing and Microstructure Symposium at the 96th ACerS Annual Meeting Indianapolis IN.
- Sept. 1994 "Advances in Ceramics Forming," International Conference on Ceramics Processing Science and Technology Friedrichshafen Fed. Rep. of Germany
- Oct. 1994 "Ceramics Manufacturing; from Science to Practice," Second International Conference on Materials Engineering for Resources Akita Japan.
- Feb. 1995 "Advanced Materials Technology: University to Commercial Practice," International Workshop on Regional Science and Technology Policy Research in Himeji (RESTPOR '95) Japan.
- March 1995 "Microstructural Order and Materials Processing for Structure and Form," University of Michigan Ann Arbor MI.
- April 1995 "Ceramic Components from Solid Free Form Fabrication Methods," International Conference on Shaping of Advanced Ceramics Mol Belgium.
- May 1995 "Chemical Aspects of Ceramic Science," Zuffanti Memorial Lecture Northeastern University Boston MA.
- July 1995 "Solid Free Form Fabrication of Ceramics." Science of Whitewares Conference Alfred University Alfred NY.
- July 1995 "Defense Science Research Council's Workshop on "Printing Technologies in Manufacturing - I&II La Jolla CA.
- March 1996 "Solution Precursor Routes to Epitaxial Films," International Symposium on Synergistic Synthesis of Inorganic Materials Tangernsee Germany.
- April 1996 "Setter Design for Binder Removal from Injection Molded Components," American Society Annual Meeting in Indianapolis Ind.
- April 1996 "Corrosion of Aluminum Nitride in Aqueous Cleaning Solutions," Materials Research Society Spring Meeting San Francisco CA.
- May 1996 "Structural Materials by 3DP," Review of ARPA/ONR Solid Freeform Program Woods Hole MA.

- June 1996 "Materials Engineering: Concepts Prototypes Processes." Science and Engineering Program for Middle and High School Teachers MIT, Camb. MA.
- Aug. 1996 "Structural Ceramic Components with Computer Derived Microstructure by Three Dimensional Printing," Solid Freeform Fabrication Symposium University of Texas at Austin, Austin TX.
- Oct. 1996 "Microstructure Order and Materials Processing for Structure and Form," Fall Quarter Seminar University of Minnesota Minneapolis MN.
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- Dec. 1996 "Rapid Prototyping and 3D Printing," American Ceramics Society and The Materials Society Chelmsford MA.
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- March 1997 "Crystallographic Texture in Films Using Ion Beams," The American Physical Society KansasCity MO.
- April 1997 "Advanced Ceramics Processing: Structure Form and Performance," University of California at Davis, Davis CA.
- April 1997 "Microstructure Order and Materials Processing for Structure and Form," Korea Advanced Institute of Technology Taejon Korea.
- May 1997 "Three-Dimensional Printing Applications and New Materials Developments," ACerS 99th Annual Meeting & Exposition Cincinnati OH.
- June 1997 "Forming and Bulk Fabrication of Ceramic Components," NSF Ceramics Workshop Washington, D.C.
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- Nov. 1997 "Advanced Materials: Concepts Prototypes and Manufacturing," University of Wisconsin-Madison Madison WI.
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- May 1998 "Anisotropic Behavior of Tape Cast Ceramic Sheets," 100th Annual Mtg. American Ceramic Society Cincinnati OH. May 3-6, 1998.
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- May 2000 "Free-Form Manufacturing of Ceramic Parts." 7th International Conference on Ceramic Processing Science, Inuyama City, Japan, May 15-8, 2000.
- Sept 2000 "Microchips as Implantable Drug Delivery Devices." Biomems & Biomedical Nanotechnology World 2000 Conference, Columbus, OH. September 23-26, 2000.
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Exhibit 2

I.D.	Heating	Low	High	P	Time	Resistance (Ohm)	Jc	Resistivity (μ Ohm-cm)
D15R60	2min to 785	50 mTorr	300	2.1	15 low/15 high	10	262500	400
D15R58	2min to 785		200	2.1	28 high	20	0	800
D15R59	2min to 785	25 mTorr	200	2.1	10 low/10 high	10	70000	400
D15R61	2min to 785	25 mTorr	200	2.1	10 low/8 high	13	112500	520
D15R56	2min to 785	26 mTorr	200	2.1	5 low/5 high	16	0	640
D15R57	2min to 785	25 mTorr	300	3	15 low/15 high	10	471250	400
D15R54	(1) 2min to 785	30 mTorr	250	3	20 low/11 high		0.3 MA/cm2	
D15R52	(1) 2min to 785	30 mTorr	200	3	15 low/15 high	9-10	0.65 MA/cm2	380
D15R50	(1) 2min to 785	30 mTorr	0	3	30 low	11-13	0.585 MA/cm2	480
D15R49	(1) 2min to 785	30 mTorr	200	3.1	15 low/33 high		0.848 MA/cm2	
D15R48	(1) 2min to 785	30 mTorr	300	1 Pure O2	15 low/15 high		1.19 MA/cm2	